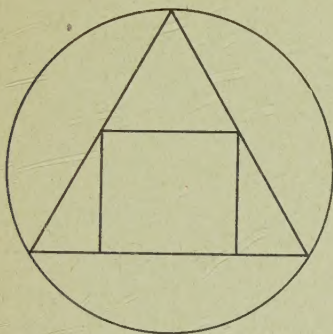


Doctor's thesis

DEPARTMENT OF PRODUCTION AND MATERIALS ENGINEERING

Lund Institute of Technology

Lund, Sweden



Influence of minor additions of reactive alloy elements on mild steel during different production heat treatments, including welding and casting, and on the resulting material properties.

NE Hannerz

Lund April 1975

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material properties.

Av

Nils Erik Hannerz

Tekn. lic., Vrml

Akademisk avhandling

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försvaras å hörsal M:B, Maskinteknik, Ole Römers väg 1,
Lund, onsdagen den 21 maj 1975 kl. 9.00.



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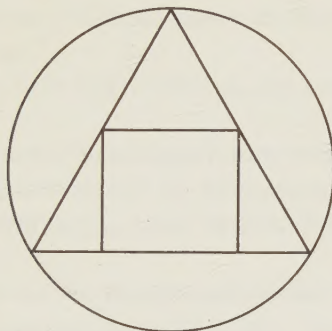
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The following papers are included in the dissertation:

- 1) "Brittleness in a cast steel caused by NbC precipitation" by NE Hannerz, U Lindberg and B Lehtinen.
Journ Iron Steel Inst 206 (1968) pp 68-73.
- 2) "Influence of cooling rate and composition on intergranular fracture of cast steel" by NE Hannerz.
Met Sc Journ 2 (1968) pp 148-152.
- 3) "Kinetics of austenite grain growth in steel" by NE Hannerz and F de Kazinczy.
Journ Iron Steel Inst 208 (1970) pp 475-481.
- 4) "Coarse grained zone. Development work with the aid of a weld simulator (in Swedish)" by NE Hannerz.
STU project 70-728/U615 . Final Report, September 1972.
- 5) "Influence of Nb on the microstructure and mechanical properties of submerged arc weld metals for CMn-Steels" by NE Hannerz, G Valland and K Easterling.
IIW report (1972) No II-612-72, XII-A-46-72, IX-798-72.
- 6) "Vanadium in mild-steel weld metal" by NE Hannerz and B M Jonsson-Holmquist. Metal Constr and Brit Weld Journ 6 (1974) pp 64-67.
- 7) "Influence of V on the weld heat affected zone properties of mild steel" by NE Hannerz and B M Jonsson-Holmquist.
Metal Science 8 (1974) pp 228-234.
- 8) "Columbium in constructional HT-steels. Influence on the heat affected zone ductility", by NE Hannerz report TD 452/74 (1974). to be published in Weld Journ 54 (1975).
- 9) "Influence of aluiminum on the HAZ notch ductility of structural steels" by NE Hannerz. Report TD 325/75 (1975).

Introduction

At the manufacture of steel structural components due considerations must be given to the alloy composition of the steel in relation to production methods including different more or less intentional heat treatments.

A steel may for instance be suited for welding involving rather rapid cooling from high temperatures (seconds or minutes), but on the other hand the very same steel composition may be prohibited in a heavy steel casting in which cooling through the high temperature regions is extremely slow (hours or days).

Some steel compositions may yield superior properties in the base material. Using high productivity welding processes yielding prolonged cooling times these properties may deteriorate completely while low productivity welding may give acceptable properties.

In the steel manufacturing process some heat treatments may be modified or even left out as a result of intentional alloying.

Thus the overall production economy from the making of the raw steel to the final fabrication of the structural component is largely dependent on steel alloy composition.

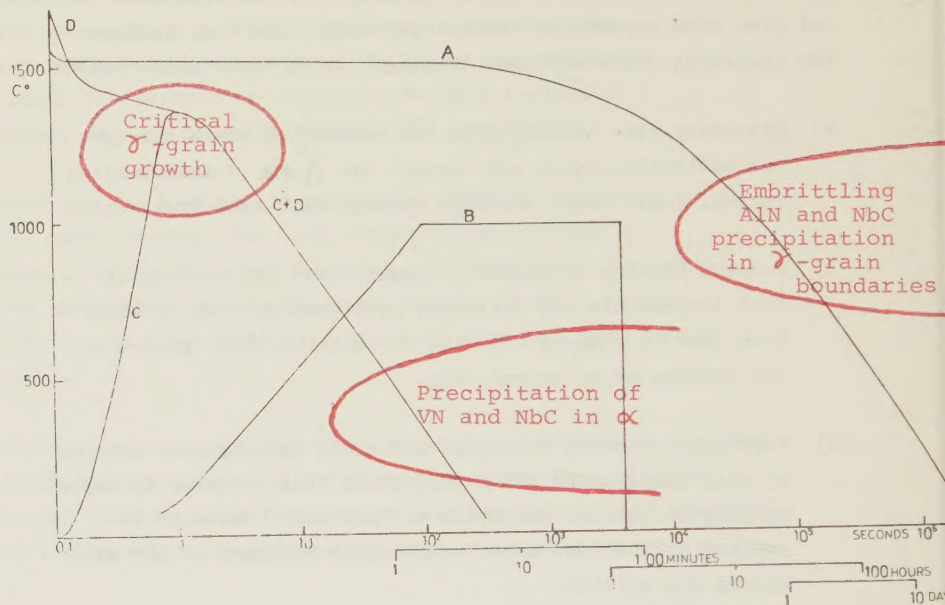
This thesis will fill out a number of white fields on the chart after which the steel man has to navigate in his search for new areas of application of the micro alloy^x technique for obtaining high strength steels with adequate properties.

X/ Document IIW-382-71 (1971) "Guide to the welding and weldability of CMn and CMn microalloyed steels" defines microalloyed steels as "steels treated with small additions of elements which are carbide-, nitride- or carbonitride- forming such as Nb, Ti, V, Al, Ta and Zr, the total content of these elements seldom exceeding 0.15%."

The work is based on studies of laboratory steel specimens but also some production scale steel heats have been studied. The following different heat treatment types have been studied:

- A) Extremely slow cooling from the solidified state through the austenite region and through the $\gamma \rightarrow \alpha$ transformation region. Heavy steel castings undergo this heat treatment.
- B) Furnace heating to austenite temperature and holding at that temperature for different prolonged periods of time. Such heat treatments are used for soaking steel prior to hot working or at normalizing.
- C) Very rapid heating to a high austenite temperature, holding at that temperature for a very short time followed by rapid cooling to ambient temperature. This heat treatment is applied to the base metal immediately adjacent to the weld during the welding.
- D) Very fast cooling from the solidified state through the austenite region and through the $\gamma \rightarrow \alpha$ transformation region. The steel weld metal is subjected to this heat treatment.

Although the cooling rate is high in cases C) and D) as compared to case A) it may be modified appreciably by changing from a high to a low energy welding process. The four heat treatment types are schematically represented overleaf.



A = cooling curve for heavy steel castings

B = heating and cooling at furnace soaking

C = heating and cooling of the heat affected zone
of the base material at welding

D = cooling curve for the steel weld metal

Paper 1 and 2. Heat treatment type A.

In paper 1, the influence of niobium on the properties of heavy steel castings has been studied. It is shown that a thin layer of NbC precipitates continuously in the austenite grain boundaries during cooling on account of the very slow cooling rates obtained. These carbides cannot be dissolved by an ordinary normalizing treatment and they will render a brittle material. This type of brittleness results in a Rock-Candy type fracture. It is a kind of intercrystalline fracture characterized by coarse grain sizes.

Paper 2 deals with the influence of aluminium and niobium on the susceptibility to this type of fracture. It is shown by cinetical and local equilibrium considerations that the cooling rate as well as the niobium and carbon contents influence precipitation of carbides and thereby the tendency to rock-candy fracture. The precipitation of AlN is analogously influenced by the aluminium and nitrogen contents as well as by the cooling rate.

Paper 3. Heat treatment type B.

Paper 3 deals with the possible use of different micro alloying elements as γ -grain refiners at furnace heat treatments in the austenite range. It is experimentally shown that vanadium, aluminium, niobium and titanium are effective in preventing grain growth. Above the temperature for the dissolution of the precipitates, the grain growth no longer is inhibited.

Also after longer holding times at desired temperatures the austenite grains may instantly grow to a much coarser size. This would be due to the coarsening of the precipitates which pin down the grain boundaries in accordance with the Zener-Hillert model.

Normal grain growth was observed as not following the $D^2 = kt$ rule first developed by Feltham. A close look at the experimental values reported for the grain growth exponent showed that the D^2 law was not followed except at very short periods of grain growth at comparatively high temperatures, in which cases the heat treatment time must in some way be corrected for the grain growth during heating and cooling. Therefore the error in the evaluated exponent may be substantial.

For aluminium, copper and steel earlier experiments with considerably prolonged soaking times had given much higher exponents ranging from 5 to 7 while the present work gave an exponent of 6.

By the, at the time, completely novel assumption that an atom in the shrinking grain must leave a kink position in this grain and jump into a kink position in the adjacent grain to allow transfer of atoms over the grain boundary from one grain to the other, it was possible to derive a formula for grain growth which better reflects experimental results.

$$D^6 - D_0^6 = kt$$

Paper 4. Heat treatment type C.

Paper 4 deals with grain growth at rapid heating to austenite temperatures and very short holding time at the peak temperatures. Carbon manganese base analysis steels were tested. It could be shown that grain growth at the highest temperatures could be restrained only with a combined addition of titanium and nitrogen. Grain refiners such as niobium, vanadium and aluminium with comparatively lower solution temperatures do not seem to restrain grain growth at these high temperatures. In paper 4 is shown furthermore that this grain refinement of the γ -grain results in a finer final grain size and improved Charpy-V toughness.

When very high peak temperatures result in a coarse δ -grain this will yield a decomposition structure characterized by proeutectoid ferrite in prior δ -grains and acicular type ferrite in the central parts of the former δ -grain. A small δ -grain will make this central zone smaller and thereby increase the toughness.

In order to understand how the δ -grain size, when decomposed in a normal way, influences the α -grain size a relationship was derived assuming that the ferrite is nucleated in the δ -grain boundaries. This relationship was tested on the different alloys yielding an equation

$$D_{\alpha} \approx 4 \sqrt[3]{D_{\delta}}$$

where 4 was obtained by experiment and the exponent was derived by the above assumption that ferrite precipitation starts in the δ -grain boundaries.

Thus refining the δ -grain will yield a refined α -grain. By using optimum additions the grain size could be decreased from 180 μm to 24 μm when the peak temperature was 1400°C.

Also, as the decreased δ -grain size gives a more abundant nucleation of ferrite, the transformation to martensite is retarded. This would yield a steel less prone to hydrogen cracking, which is postulated in paper 4.

Paper 5 and 6. Heat treatment type D.

Paper 5 shows what happens during cooling in weld metal a steel containing niobium. The heat treatment cycle is identical with that of heavy steel castings except for the cooling rate which in the weld is faster by several orders of magnitude. No grain boundary precipitation in the δ -region occurs.

It is evident, however, that coherent niobium carbo-nitrides or carbo-nitride zones precipitate in the ferrite. The carbo-nitrides harden the lattice which results in increased brittle ductile transition temperature. It has been possible to see some precipitates in the electron microscope but evidently the particles are on the verge or below the resolving power of the electron microscope.

In paper 6 it is shown that also in vanadium bearing welds carbo-nitrides precipitate in the ferrite on cooling, which gives a less ductile weld on account of precipitation hardening.

Stress relieving heat treatment which is stipulated by different societies gives a still further increased vanadium precipitation. The embrittlement is a function of vanadium and niobium content. The vanadium carbo-nitrides are more easy to see in the electron microscope than the niobium carbo-nitrides, presumably due to the coarser particles and a more abundant precipitation.

Paper 7, 8 and 9. Heat treatment type C.

Paper 7 deals with the influence of vanadium on the heat affected zone properties at welding. At the very high peak temperatures obtained all vanadium nitrides dissolve into solid solution of vanadium and nitrogen in the austenite to reprecipitate in α -phase in the same way as in the weld metal at cooling to ambient temperature.

Paper 8 deals with the analogous phenomenon in the heat affected zones of niobium treated steels. The precipitation of niobium and vanadium carbo-nitrides in the heat affected zones renders this zone brittle compared to the unalloyed material. The mechanism is the same as in the weld metal. The precipitates are of the same size i.e. the vanadium carbo-nitrides are somewhat easier to discern with the aid of an electron microscope than the niobium carbo-nitrides

some of which are of sizes on the very verge of the resolving power of the electron microscope most of which lie below. The embrittlement increases with rising vanadium as well as niobium contents.

Slower cooling rates, as obtained by high energy electro-gas welding, gives a coarser or a more abundant precipitation and hence a more severe embrittlement than do low energy welding methods.

In paper 9 is shown that aluminium-treated steels do not behave in a similar manner. No brittleness caused by the dispersed precipitation of aluminium nitride has been traced, although two different cooling rates after weld simulation were tested. This is explained by the fact that AlN precipitation in ferrite is an extremely sluggish reaction at the low temperatures in question, why no embrittling precipitation takes place. Also there is no case reported in the literature where AlN in a steel has been used as precipitation hardener. It is quite obvious that the aluminium-treated steels in this respect differs from niobium-, vanadium- and titanium-treated steels (n.b. if titanium in respect of nitrogen content is hyperstoichiometric in which case TiC may precipitate).

In paper 9, as well as in paper 4, further, the detrimental effect of free nitrogen on HAZ toughness is demonstrated.

Summary and conclusions

The influence of microalloy elements on steel properties has been investigated.

It has been shown that niobium and also aluminium may cause brittleness on extremely slow cooling from high temperatures in heavy steel castings.

At very rapid cooling as encountered in steel welds and weld heat affected zones vanadium and niobium may cause brittleness.

Titanium on the other hand may improve the toughness of the heat affected zone as well as the resistance to hydrogen cracking.

Also the kinetics of grain growth at furnace soaking was studied.

The results obtained are in accordance with what may be expected on the bases of physical metallurgical reasoning, which is supported by cinetical calculations assuming local equilibrium conditions.

By setting down limits and in some cases by advancing the frontier for the successful exploitation of the microalloy technique this thesis may be seen as an essential contribution to structural steel research.

